

# PATENT SPECIFICATION

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## COMPLETE SPECIFICATION.

### Process for the Improvement of Lacquers.

We, DEUTSCHE GOLD-UND SILBER-SCHEIDEANSTALT VORMALS ROESSLER, of 9 Weissfrauenstrasse, Frankfurt (Main) 1, Germany, a Body Corporate organised

5 under the laws of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 This invention relates to a process for the improvement of lacquers by introducing finely divided silicon dioxide into lacquers, which process is particularly suitable for the handling of highly active silicon dioxide which has been pyrogenically produced by decomposition of volatile silicon compounds in the vapour phase.

15 It is known that pyrogenically obtained silicon dioxide can advantageously be used, in comparatively small quantities, in lacquers as a substance for delaying sedimentation or as a matting agent. This oxide, which consists predominantly of particles of about 5 to  $100 \times 10^{-6}$  mm. and has a large specific surface, is not easily incorporated into liquids, because, on the one hand, it has a low bulk density and, on the other hand, it forms light dust. Consequently, it

20 is frequently necessary to combine small quantities of pyrogenic silicon dioxide with the lacquer on a roll stand in order to obtain a homogeneous distribution with uniform wetting.

25 It has now been found that these difficulties experienced when using pyrogenic silicon dioxide, which is of great interest in the lacquer industry, can effectively be avoided if the finely-divided silicon dioxide is processed to form a liquid dispersion with a high solids content which amounts to at least 25% by weight but preferably 30—45% by weight and this dispersion is then added

30 to the lacquer, the dispersing agent having a predominantly polar character. The result hereby obtained is that all the quantity of lacquer does not have to be processed on the roll stand, since the distribution of the dispersion in the lacquer can be effected in an extremely short time by stirring. The production of the dispersion from the finely divided silicon dioxide and the liquid dispersing agent can on the other hand be achieved without any appreciable labour expense though the dispersion itself must be produced by trituration on a roll stand.

35 The process of the present invention is based on the surprising discovery that certain liquids, in spite of the known strong thickening action of finely divided silicon dioxide are unexpectedly able to take up large quantities of this solid without losing their liquid character. It is however necessary for this purpose to use certain dispersion liquids, which must not only be compatible with the lacquer constituents, but must above all have a polar character, i.e. comprise predominantly hydrophilic groups. Whereas non-polar liquids have only a comparatively small absorption power for pyrogenically obtained silicon dioxide, and are not suitable as dispersing agents for the purposes of the present invention, liquids having polar and non-polar groups in the molecule have unexpectedly proved to be quite suitable. The same also applies as regards mixtures of polar and non-polar substances which, in the same way as the liquids mentioned above can readily absorb 40 to 50% of their weight of the solid without any thickening into pastes or even pseudo-solid elements. Some of the dispersing agents suitable for the purposes of the invention are set out below in Table I this table indicating their absorption capacity for pyrogenically obtained silicon dioxide with a par-

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particle size of  $5$  to  $25 \times 10^{-4}$  mm. and a specific surface of about  $200 \text{ m}^2/\text{g}$ . All dispersions are liquids.

TABLE I

	Dispersing agent	Silicon dioxide % by weight
5	Methanol	39
10	Ethanol	31
	<i>n</i> -propanol	42
	<i>n</i> -butanol	45
	<i>i</i> -propanol	37
	<i>i</i> -butanol	40
15	Isanol (60% <i>n</i> -butanol + 40% <i>i</i> -butanol) by weight	43
	Ethylene glycol monomethyl ether	38
	Ethylene glycol monobutyl ether	42
20	Toluene	11
	Benzene	11
	Benzine (b.p. $<140^\circ \text{ C}$ )	12

Toluene, benzene and benzine are examples of non-polar liquids in the last part of the table for comparison with the polar liquids, and it will be seen from the table how great the difference is as regards the absorption power of these two types of liquids.

The following table reproduces corresponding experiments which were carried out with mixtures of polar and non-polar liquids, on a sample of pyrogenic silicon dioxide different from that used in the experiments reported in Table I but of similar particle size.

TABLE II.

	Dispersing agent	Silicon dioxide %
35	Ethanol	28
40	Ethanol/benzene, 90/10 by weight	34
	Ethanol/benzene, 90/10 by weight	38
	Ethanol/toluene, 90/10 by weight	36
45	<i>i</i> -propanol	33
	<i>i</i> -propanol/benzene, 90/10 by weight	41
	<i>i</i> -propanol-benzene, 85/15 by weight	38
50	<i>i</i> -propanol/toluene, 85/15 by weight	43
	Benzine (b.p. $<140^\circ \text{ C}$ )	9
	Benzene	11
	Toluene	11

55 The table shows that the use of dispersing agents consisting of a mixture of polar and non-polar liquids enables the maximum absorption power for finely divided silicon dioxide to be raised far beyond the values which are to be expected additively.

One particular advantage of the above described dispersions with a high solid content is that these dispersions unexpectedly have an extremely favourable storage capacity. For example, should a dispersion of 40% by weight of pyrogenic silicon dioxide in *n*-butanol be stored for 5 weeks in a container having a cover which is a tight fit, the dispersion is only slightly thickened and can readily be liquefied by stirring for a short time. Another dispersion of similar composition was still completely liquid under the same conditions, after the same storage time.

70 This property of the dispersions used according to the invention was also verified by viscosity measurements. It was found that both the viscosity itself, and also the thixotropic properties of the highly concentrated dispersions, had remained almost completely unchanged over a period of 3 weeks, so that even after this storage period, the said dispersions could be used immediately for introducing fillers into lacquers.

75 For carrying the process of the invention into practice, it has proved to be particularly advantageous for the dispersion not to be added directly to the lacquer, but for it to be introduced by simple stirring into diluted lacquer and only then to combine the latter with the lacquer. This produces a particularly simple and uniform distribution of the silicon dioxide in the lacquer, this leading to a matting which is completely free from spots when the lacquer is employed.

80 The invention is further illustrated by the following example:—

## EXAMPLE.

85 A 35% by weight dispersion of a finely divided pyrogenic silca, known under the Registered Trade Mark "Aerosil", in *n*-butanol was prepared by grinding on a roll stand. The composition of the nitro-cellulose lacquer to be matted was as follows:—

	Parts by weight
Toluene	306
<i>n</i> -butyl acetate, 85%	150
Ethyl acetate	122
<i>n</i> -butanol	31
Collodion cotton (moist with butanol)	262
Collodion cotton (dry)	170
Dibutyl phthalate	38
Modified urea resin (Uresin B)	88

90 Used as diluent was a mixture consisting of:—

	Parts
Toluene	306
<i>n</i> -butyl acetate (85%)	156
Ethyl acetate	122
<i>n</i> -butanol	31

The dispersion prepared as initially described was incorporated by stirring in a quantity of 5.3 g., corresponding to a solid content of 1.85 g. of silicon dioxide, into a 5 dilution of the lacquer, using a high-speed stirrer mechanism and 80 parts by weight of this diluted lacquer were mixed with 100 parts by weight of lacquer. There was obtained a lacquer having exceptionally good 10 matting power, in which sedimentation or demixing phenomena did not occur over a period of 4 weeks.

**WHAT WE CLAIM IS:—**

- 15 1. A process for the improvement of lacquers by introducing finely divided silicon dioxide prepared pyrogenically, into lacquers, wherein the finely divided silicon dioxide is added to the lacquer in the form of a liquid dispersion with a solid content of at least 25% by weight in a dispersing agent with a predominantly polar character.
- 20 2. A process as claimed in Claim 1, wherein the solid content of the dispersion is 30 to 45% by weight.

3. A process as claimed in Claim 1 or 25 2, wherein the dispersing agent contains polar and non-polar groups.

4. A process according to Claim 1 or 2, 30 wherein the dispersing agent is a mixture of polar and non-polar substances.

5. A process as claimed in any of Claims 1 to 4, wherein the silicon dioxide dispersion is added to diluted lacquer and this mixture is then added to the lacquer.

6. A process for the improvement of 35 lacquers substantially as described with reference to the example.

7. Improved lacquers whenever produced by the process claimed in any of the preceding claims. 40

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